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(19) (CA) **APPLICATION FOR CANADIAN PATENT** (12)

(54) Oil Emulsion

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Notice: This application is as filed and may therefore contain an incomplete specification.



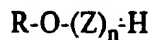
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Abstract

The oil emulsions comprise

(a) at least one alkyl polyglycoside of the general formula

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where the radical R is a linear or branched saturated or unsaturated alkyl radical with 8 to 16, preferably 8 to 12, carbon atoms, the radical $-(Z)_n-H$ is a polyglycosyl radical, Z is a glycosyl radical, and
15 n has an average value of 1 to 5,

(b) at least one oil,

(c) water,

where the ratio of component (a) to component (b) is 1.5:1 to 15:1, preferably 2.5:1 to 10:1, by weight and to the use of C_8/C_{10} -alkyl
20 polyglycosides as emulsifiers for oils.

Oil emulsion

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The invention relates to oil emulsions in water or water/alcohol mixtures, preferably of essential oils, which can be employed in cosmetics or pharmaceuticals.

DE-A1-41 10 506 discloses emulsifiers for preparing oil-in-water
10 emulsions of essential oils which can be used in cosmetics or medicine. The emulsifiers used are C_{10}/C_{12} - and C_{12}/C_{14} -alkyl polyglucosides. The maximum ratio of alkyl polyglucoside to oil is 1:4.

DE-A1-40 33 928 describes oil-in-water emulsions which contain alkyl polyglycosides. C_{14} -, C_{16} - and C_{18} -alkyl glucosides are described. The
15 maximum ratio by weight of alkyl polyglucoside to oil is 1:3. The oil emulsions furthermore contain a fatty acid partial glyceride as crystallization inhibitor.

EP-A1-0 418 479 describes emulsifiers for preparing storage-stable, aqueous polysiloxane or polysiloxane/liquid paraffin emulsions. C_{10}/C_{12} -,
20 C_{12}/C_{13} - and C_{12}/C_{14} -alkyl polyglycosides are used, with the maximum ratio by weight of oil to alkyl polyglycoside being 4:1.

It is an object of the present invention to provide oil emulsions which are toxicologically unobjectionable.

Another object of the present invention is to provide oil emulsions
25 which are clear and stable.

Another object of the present invention is to provide oil emulsions which have a good foaming capacity.

We have found that these objects are achieved by an oil emulsion as described in the claims and by the use of the alkyl polyglycosides
30 according to the invention.

It has been found according to the invention that the oil emulsions from water-in-oil mixtures obtained on use of alkyl polyglycosides as emulsifiers have a clear appearance, a high stability and a high foaming capacity.

5 The alkyl polyglycosides employed according to the invention may, where appropriate, be used with other coemulsifiers which are nonionic, cationic or anionic surfactants. The alkyl polyglycosides are known to be compatible with skin and to be well tolerated by the skin and mucous membranes. They are toxicologically unobjectionable.

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Alkyl polyglycosides

The term "alkyl polyglycosides" means the products of the reaction of sugars and aliphatic alcohols. The alkyl polyglycosides employed according to the invention can generally be described by the formula $R-O-(Z)_n-H$ 15 where the radical R is a linear or branched, saturated or unsaturated C_{8-16} -alkyl radical, preferably C_{8-12} -alkyl radical. The radical R is preferably a straight-chain, saturated alkyl radical with 8-10 carbon atoms, and a particularly preferred alkyl polyglycoside has a ratio of C_8 - to C_{10} -alkyl radicals of 0.3:1 to 1:0.3.

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The radical $-(Z)_n-H$ is a polyglycosyl radical, ie. a monovalent radical from n glycosyl radicals which are preferably linked linearly, preferably a reducing saccharide residue. The polyglycosyl radical can be an oligo- or monoglycosyl radical. Examples of sugar components which can be used as alkylglycosyl radicals are glucose, fructose, mannose, galactose, 25 talose, gulose, allose, altrose, idose, arabinose, ribose, maltose, lactose, maltotriose, dextrose, starch. It is likewise possible to employ mixtures of these sugars. The preferred polyglycosyl radical is the glucosyl radical. The polyglycosyl radical $-(Z)_n-H$ is preferably an oligosaccharide or a monosaccharide, preferably a hexose or pentose, in particular glucose.

The alkyl radical can be bonded in the form of an acetal to more than one glucose residue, ie. it can be bonded to a poly- or oligosaccharide residue.

The aliphatic primary alcohols can be natural fatty alcohols which are prepared, for example, by hydrogenation of fatty acids. It is likewise possible to employ synthetic primary alcohols such as oxo alcohols or Ziegler alcohols. Straight-chain alcohols are preferably employed, such as n-octanol, n-decanol, n-dodecanol and mixtures thereof.

The alkyl polyglycosides are, as a rule, mixtures which, besides small amounts of residual alcohol, also contain unreacted monosaccharides, oligosaccharides or oligoalkyl polyglycosides.

n describes the average number of polyglycosyl radicals per alcohol residue and is also referred to as the degree of condensation. The average value of n is from 1 to 5, preferably from 1 to 1.3.

The alkyl polyglycosides employed according to the invention can be prepared by known processes as described, for example, in DE-A 42 12 080 or else in EP-A1-0 306 650, EP-A1-0 306 652, EP-A1-0 306 651 or EP-A2-0 448 799. They can be linked directly by reacting the long-chain alcohol component with the sugar component with elimination of water. However, they can also be prepared by the transacetalization process, in which case a short-chain alkyl glycoside is initially synthesized as intermediate and is then converted in a second stage by transacetalization with long-chain alcohols into the required alkyl polyglycoside.

The oil emulsions according to the invention are probably microemulsions of oil in water or water/alcohol mixtures.

It is particularly preferred according to the invention for the C₈/C₁₀-alkyl polyglycosides which have been prepared according to one embodiment of the invention by the process described in DE-A-42 10 080 to be used as emulsifiers for oils, especially essential oils.

Alkyl polyglycosides which can be employed according to the invention can also be mixtures of different alkyl polyglycosides which are prepared by mixing alkyl polyglycosides with alkyl monoglycosides.

Coemulsifiers

5 According to one embodiment of the invention, the oil emulsions can additionally contain coemulsifiers. These coemulsifiers are, for example, nonionic, cationic or anionic surfactants. The amount of coemulsifier employed can be, based on the alkyl polyglycoside content, from 0 to 15% by weight, preferably 0 to 10% by weight.

10 Examples of coemulsifiers which can be used are fatty alcohol sulfates, fatty alcohol ether sulfates, alkane sulfonates, fatty alcohol ethoxylates, fatty alcohol phosphates, fatty alcohol ether sulfonates, alkyl betaines, sorbitan esters, POE-sorbitan esters, sugar fatty acid esters, fatty acid polyglycerol esters, fatty acid partial glycerides, fatty acid carboxylates, fatty
15 alcohol sulfosuccinates, fatty acid sarcosinates, fatty acid isethionates, fatty acid taurinates, citric esters, silicone copolymers, fatty acid polyglycol esters.

Oil

20 Any desired oils can be used in the oil emulsions according to the invention. Essential oils are preferably used according to one embodiment of the invention. Examples of essential oils which can be used are larch oil, lavender oil, rosemary oil, pine needle oil, pine oil, eucalyptus oil, peppermint oil, sage oil, bergamot oil, turpentine oil and mixtures thereof. Other
25 suitable essential oils are known to the skilled worker.

Other ingredients

The oil emulsions according to the invention may contain other ingredients, for example alcohols. The alcohol preferably used in this case is a non-toxic alcohol which is tolerated by skin and is miscible with water,

such as ethanol or isopropanol. Other suitable alcohols may likewise be used.

The oil emulsion according to the invention may also be free of alcohol.

5 Other ingredients which can be incorporated into the oil emulsions according to the invention are, for example, electrolytes, dyes, preservatives, acids such as lactic acid or citric acid, or bases.

Preparation of the oil emulsions

According to a preferred embodiment of the invention, the chosen
10 alkyl glycoside is initially intimately mixed, for example using a stirrer, with the particular essential oil or perfume oil used, in the required amounts. This mixing is preferably carried out at room temperature. Demineralized water is then introduced slowly while stirring continuously. In
15 order to define the most suitable amounts of the alkyl glycoside in each case to achieve an optimal solubilizing effect it is possible to use turbidimetric titration as described, for example, by A. Domsch, "Die kosmetischen Präparate", 4th edition of the work founded by G.A. Novak, volume II "Wäßrige und tensidhaltige Formulierungen".

According to another embodiment of the invention, the alkyl
20 glycoside is initially dissolved or dispersed in the aqueous phase and subsequently the oil is added with stirring.

The ratio by weight of alkyl glycosides to oil or oils is moreover 1.5:1 to 15:1, preferably 2.5:1 to 10:1, in particular 3:1 to 6:1. The alkyl polyglycoside used, preferably the C₈/C₁₀-alkyl polyglycoside, in particular
25 the C₈/C₁₀-alkylpolyglucoside, can moreover be employed as a solid substance or as an aqueous solution. The amounts of substances used according to one embodiment of the invention are 3 to 6 g (based on the active substance) of the required alkyl glycoside, 1 g of the oil used, preferably essential oil or perfume oil, and demineralized water ad 100 g.

The oil emulsions according to the invention have a good foaming capacity, are toxicologically unobjectionable and clear and stable systems.

According to a preferred embodiment, C_8/C_{10} -alkyl polyglycosides, in particular C_8/C_{10} -alkyl glucosides, are employed as emulsifiers for essential oils.

Determination of the foam height

To measure the foaming capacity and the foam stability, the foam height (foam index) is determined for the oil emulsions according to the invention.

This entails a mixture of alkyl polyglycoside and oil being prepared and 1 g of this mixture being introduced into a tall-form graduated beaker with a capacity of 1 l. Then 600 g of demineralized water are introduced. The contents of the beaker are then mixed or stirred at room temperature using an electric stirrer consisting of an IKA motor and a paddle agitator, which is arranged at the level of the 200 ml mark in the beaker, at a speed of about 1400 rpm.

After stirring for 1 minute, the stirrer is switched off and, after 30 seconds, the foam height is measured (in cm). The stirrer is then started again, and stirring is continued for 3 minutes at the same speed. After waiting once again for 30 seconds, the foam height is measured again (in cm). The measured foam heights (in cm) indicate the foam index in each case.

It is also possible to employ in place of water a water/alcohol mixture, for example 600 g of a water/ethanol mixture with a water to ethanol ratio of 80:20 by weight.

The invention is explained in detail hereinafter by means of examples.

Examples A1 to A6 show the properties of the oil emulsions according to the invention. The emulsions are clear and stable for more than one week. The foam values for these formulations are higher than for

the corresponding comparative examples (comparative examples B1 to B4 compared with examples A1 to A4). An alkyl polyglucoside used according to the invention was employed in comparative examples B1 to B6, the alkyl polyglucoside to essential oil ratio being 1:1 by weight. In contrast to the
5 formulations according to the invention, these formulations have a cloudy or milky appearance and are not stable for long, ie. less than 24 hours.

A commercial solubilizer (an ethoxylated hydrogenated castor oil with the name Cremophor RH40) was employed in comparative example B7 to B10. The appearance of the formulations is cloudy, and they show only
10 low foam values.

Example A1

1 g of rosemary oil and 3 g of C₈/C₁₀-alkyl polyglucoside (degree of condensation 1.2) are mixed with 96 g of water and stirred at room temperature for 5 minutes. The result is a clear solution which is stable for
15 a long time, ie. more than one week.

Example A2

1 g of lavender oil and 3 g of C₈/C₁₀-alkyl polyglucoside (degree of condensation 1.2) are mixed with 96 g of water and stirred at room temperature for 5 minutes. The result is a clear solution which is stable for
20 a long time, ie. more than one week.

Example A3

1 g of pine needle oil and 3 g of C₈/C₁₀-alkyl polyglucoside (degree of condensation 1.2) are mixed with 96 g of water and stirred at room temperature for 5 minutes. The result is a clear solution which is
25 stable for a long time, ie. more than one week.

Example A4

1 g of pine needle oil and 3 g of C₈/C₁₀-alkyl polyglucoside (degree of condensation 1.2) are mixed with 76.8 g of water and 19.2 g of ethanol and stirred at room temperature for 5 minutes. The result is a clear
30 solution which is stable for a long time, ie. more than one week.

Example A5

1 g of lavender oil and 3 g of C_8/C_{10} -alkyl polyglucoside (degree of condensation 1.2) are mixed with 76.8 g of water and 19.2 g of ethanol and stirred at room temperature for 5 minutes. The result is a clear solution which is stable for a long time, ie. more than one week.

Example A6

1 g of the oral hygiene flavoring oil Dragoco ZM0065 and 3 g of C_8/C_{10} -alkyl polyglucoside (degree of condensation 1.2) are mixed with 76.8 g of water and 19.2 g of ethanol and stirred at room temperature for 5 minutes. The result is a clear solution which is stable for a long time, ie. more than one week.

The foam indices were determined after one minute and three minutes for the solutions of examples A1 to A4. They are listed in Table I and II.

Comparative examples

Example B1

1 g of rosemary oil and 1 g of C_8/C_{10} -alkyl polyglucoside (degree of condensation 1.2) are mixed with 98 g of water and stirred at room temperature for 5 minutes. The result is a very cloudy solution.

Example B2

1 g of lavender oil and 1 g of C_8/C_{10} -alkyl polyglucoside (degree of condensation 1.2) are mixed with 98 g of water and stirred at room temperature for 5 minutes. The result is a milky liquid which separates into two phases after standing for 24 hours.

Example B3

1 g of pine needle oil and 1 g of C_8/C_{10} -alkyl polyglucoside (degree of condensation 1.2) are mixed with 98 g of water and stirred at room temperature for 5 minutes. The result is a milky liquid which separates into two phases after standing for 24 hours.

Example B4

1 g of pine needle oil and 1 g of C₈/C₁₀-alkyl polyglucoside (degree of condensation 1.2) are mixed with 78.4 g of water and 19.6 g of ethanol and stirred at room temperature for 5 minutes. The result is a very cloudy solution which becomes milky after standing for 24 hours.

Example B5

1 g of lavender oil and 1 g of C₈/C₁₀-alkyl polyglucoside (degree of condensation 1.2) are mixed with 78.4 g of water and 19.6 g of ethanol and stirred at room temperature for 5 minutes. The result is a milky solution which separates into two phases after standing for 24 hours.

Example B6

1 g of the oral hygiene flavoring oil Dragoco ZM0065 and 1 g of C₈/C₁₀-alkyl polyglucoside (degree of condensation 1.2) are mixed with 78.4 g of water and 19.6 g of ethanol and stirred at room temperature for 5 minutes. The result is a milky solution.

The commercial solubilizer Cremophor RH40 was employed in place of the C₈/C₁₀-alkyl polyglucoside in the following comparative examples B7 to B10.

Example B7

1 g of rosemary oil and 3 g of Cremophor RH40 are mixed with 96 g of water and stirred at room temperature for 5 minutes. The result is a very cloudy solution.

Example B8

1 g of rosemary oil and 1 g of Cremophor RH40 are mixed with 98 g of water and stirred at room temperature for 5 minutes. The result is a very cloudy solution.

Example B9

1 g of pine needle oil and 3 g of Cremophor RH40 are mixed with 96 g of water and stirred at room temperature for 5 minutes. The result is a very cloudy solution.

Example B10

5 1 g of pine needle oil and 1 g of Cremophor RH40 are mixed with 98 g of water and stirred at room temperature for 5 minutes. The result is a very cloudy solution.

The foam indices were determined after one and three minutes for the formulations of comparative examples B1 to B4 and B7 to B10. They
10 are listed in Tables I and II.

TABLE I
Foam indices in water

Example	Emulsifier or solubilizer	Oil employed	Mixing ratio	Foam index 1 min/ 3 min
A1	C ₈ -C ₁₀ glucoside	Rosemary oil	3(AS):1	2.2/4.2
B1	C ₈ -C ₁₀ glucoside	Rosemary oil	1(AS):1	1.9/3.8
A2	C ₈ -C ₁₀ glucoside	Lavender oil	3(AS):1	2.2/4.6
B2	C ₈ -C ₁₀ glucoside	Lavender oil	1(AS):1	1.6/2.9
A3	C ₈ -C ₁₀ glucoside	Pine needle oil	3(AS):1	2.0/4.0
B3	C ₈ -C ₁₀ glucoside	Pine needle oil	1(AS):1	1.4/2.0
B7	Cremophor RH 40	Rosemary oil	3(AS):1	1.2/2.1
B8	Cremophor RH 40	Rosemary oil	1(AS):1	0.6/0.9
B9	Cremophor RH 40	Pine needle oil	3(AS):1	1.0/1.5
B10	Cremophor RH 40	Pine needle oil	1(AS):1	0.5/0.6

TABLE II

Foam indices in ethanol:water - ratio 20:80 by weight

Example	Emulsifier	Oil employed	Mixing ratio	Foam index 1 min/ 3 min
A4	C ₈ -C ₁₀ glucoside	Pine needle oil	3(AS):1	2.2/1.9
B4	C ₈ -C ₁₀ glucoside	Pine needle oil	1(AS):1	1.5/2.0

It is evident from the examples that the oil emulsions according to the invention are clear and stable for a long time and have a good foaming

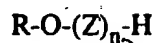
capacity. The alkyl polyglycosides according to the invention can thus be used to prepare clear emulsions of oil in water or water/alcohol mixtures with high stability and high foaming capacity. In contrast to these, the mixtures in the comparative examples have a poorer foaming capacity. They
5 are cloudy or milky liquids which, in some cases, separate into two phases.

The emulsions according to the invention can be employed, for example, in cosmetics or in pharmaceutical applications, for example in bath oils, shaving lotions, face lotions, mouthwashes, hair lotions. Oil emulsions
10 which contain alcohol can be used as eau de cologne or eau de toilette.

5 We claim:

1. An oil emulsion comprising

(a) at least one alkyl polyglycoside of the general formula



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where the radical R is a linear or branched saturated or unsaturated alkyl radical with 8 to 16, preferably 8 to 12, carbon atoms, the radical $-(Z)_n-H$ is a polyglycosyl radical, Z is a glycosyl radical, and n has an average value of 1 to 5,

15 (b) at least one oil,

(c) water,

where the ratio of component (a) to component (b) is 1.5:1 to 15:1, preferably 2.5:1 to 10:1, by weight.

2. An oil emulsion as claimed in claim 1, wherein the ratio of component (a) to component (b) is 3:1 to 6:1 by weight.

20 3. An oil emulsion as claimed in claim 1, which additionally comprises (d) at least one alcohol which is miscible with water.

4. An oil emulsion as claimed in claim 1, which is free of alcohol.

5. An oil emulsion as claimed in claim 1, wherein the alkyl polyglycoside is a C₈/C₁₀-alkyl polyglycoside.

25 6. An oil emulsion as claimed in claim 5, wherein the molar ratio of C₈- to C₁₀-alkyl radicals is 0.3:1 to 1:0.3.

7. An oil emulsion as claimed in any of claims 1 to 4, wherein n has an average value of from 1 to 1.3.

30 8. An oil emulsion as claimed in any of claims 1 to 4, wherein the polyglycosyl radical $-(Z)_n-H$ is an oligosaccharide or a monosaccharide, preferably a hexose or pentose, in particular glucose.

9. An oil emulsion as claimed in any of claims 1 to 4, wherein the oil is an essential oil.
10. An oil emulsion as claimed in any of claims 1 to 4, which additionally comprises (e) at least one coemulsifier which is a nonionic, cationic or
5 anionic surfactant.
11. An oil emulsion as claimed in any of claims 1 to 4, which is clear.
12. The use of alkyl polyglycosides as described in claim 5, as emulsifiers for oils, in particular essential oils.
13. The use of alkyl polyglycosides as described in any of claims 1 to 4
10 for preparing clear emulsions of oil in water or water/alcohol mixtures.
14. The use of the oil emulsion as claimed in any of claims 1 to 4 in bath oil, shaving lotion, face lotion, mouthwash or hair lotion.